A transmission electron microscope and X-ray diffraction study of muscovite and chlorite

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SUMMARY. Single-crystal diffraction patterns produced by transmitted radiation, both X-rays and electrons, reveal varying degrees of disorder and long-range order in common phyllosilicates from several different rock types. The transmission electron micrographs and their selected-area diffraction patterns demonstrate the presence of numerous stacking faults parallel to (001) of muscovite and chlorite. Individual stacking faults can be recognized by the diffraction-contrast fringe patterns they cause, and partial dislocations can be seen where such faults terminate inside a crystal. Long-range order of muscovite explains what seemed to be spurious, high 'background' levels that are sometimes encountered in the analysis of rock fabrics by transmitted X-rays.

IF we depart from the usual method of obtaining an X-ray diffractogram from a powder sample and, instead, transmit X-rays through a slice of the whole rock ground to a thickness of approximately 100 μm (Oertel, 1970), we would expect several differences from the usual results. Absorption of X-rays in the thick sample should reduce the intensity of diffracted rays and worsen the counting statistics, the inability to use para-focusing geometry should result in broad peaks and difficulties in the discrimination of crystal planes with small differences of d-values, and there should be a strong influence on the relative size of peaks from the same mineral of any preferred orientation of that mineral. All these differences are, in fact, found when transmitted X-rays are used to study rock fabrics. In addition, however, an unexpected phenomenon was noted in transmission X-ray diffractograms of some slates rich in muscovite having a strong preferred orientation of the basal plane parallel to the slaty cleavage. Diffractograms of such slates appropriately oriented to enhance the $00l$ reflections of muscovite frequently show unusually high 'background' levels at 2θ values intermediate between the $00l$, $l = 2n$ reflections. In some specimens this 'background' appears most intense at 2θ for $00l$, l odd, that is, where diffraction is forbidden for a mineral with the space group $C2/c$.

When a single crystal of muscovite, the powder of which gives a 'normal' X-ray diffractogram with no evidence of any violation of $C2/c$ symmetry, was cut into a sample normal to the basal plane and a diffractogram of it made in transmission, the existence of 'forbidden' reflections, evidence for long-range order, was confirmed.

The recent perfection of the ion-bombardment thinning technique allowed preparation of ultrathin samples of a slate and of a carbonate-cemented nodule in a shale for

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transmission electron microscopy. With this method, electron-diffraction patterns of selected grains or assemblages of grains can be produced, and these patterns can be compared with X-ray diffractograms. Evidence for varying degrees of disorder was found in muscovite and chlorite. Forbidden spots were found in the diffraction patterns of muscovite.

Previous work. Layer silicates, including the micas, chlorites, and clays, are known to form numerous polymorphs (Bragg and Claringbull, 1965; Verma and Krishna, 1966). Brindley *et al.* (1950) first noted that diffuse streaks along X-ray reflections in rotation photographs of chlorite were due to random rather than orderly stacking of layers. That similar polytypes also exist in micas was shown by Hendricks and Jefferson (1939).

Smith and Yoder (1956) demonstrated that the silicon–oxygen layers of mica facing each other across the octahedral layer can be stacked with an angle of stagger of $n\pi/3$, where n is an integer including zero. Allowing only these angles of stagger in simple alternations, the various polytypes can be derived. Polytype $2M_1$, in which n alternates between 2 and 4, is by far the most common of the polytypes of muscovite. Radoslovich (1959) and Zvyagin (1962) have re-examined the structural control of the polytypes of mica. All this work is based on the various techniques of registering X-ray diffraction by single crystals or powder of crystal fragments.

Until recently transmission electron microscopy on phyllosilicates has been restricted to work on plates cleaved along (001). This does not permit [001] to be tilted far from the direction of the incident beam to study the $00l$ reflection. As a consequence, relatively little work on the stacking structure of phyllosilicates has been done with this technique. Levinson (1953) studied muscovite and lepidolite but could provide no information on order along the *c*-axis. Eckhardt (1958) observed more diffraction spots (other than $00l$) on a pattern from a muscovite flake than expected for an ordered $2M$ muscovite. He tentatively explained their presence by a superlattice, considering the flake as a single crystal, but he could not exclude the possibility of double diffraction by two individuals. Superlattice reflections, caused by long-range order, are a well-known effect in alloys. Many examples are cited in a review by Sato and Toth (1965). However, no such work has been done on phyllosilicates.

The difficulty of not being able to observe phyllosilicate crystals with the basal plane parallel to the incident beam in transmission electron microscopy has recently been overcome. Using the ion-bombardment technique we succeeded in thinning rock slices into ultrathin electron-transparent sections that include phyllosilicate grains with the basal plane at right angles with the specimen plane. Preliminary studies (Phakey *et al.*, 1972) revealed that $00l$ diffraction spots of such minerals are frequently extensively streaked and that in some cases 'forbidden' $00l$ diffraction spots are present.

Specimens. Powder and transmission X-ray diffractograms were prepared from a large single crystal of muscovite (University of California, Los Angeles, Department of Geology Specimen MS 2715, collected by W. J. Miller from a pegmatite dyke 5·5 km SSE. of Chesterton, New York).

Transmission electron micrographs of chlorite and muscovite were obtained from a Middle Cambrian purple slate from a quarry near Nantlle, Caernarvon, North Wales

(Sample W22 from the Dorothea Slate Quarry, National Grid Reference SH 249 850, 353 200). The rock consists of strongly oriented muscovite and chlorite together with quartz and minor albite and hematite. The texture of the rock has been described by Oertel and Phakey (1972) and by Phakey *et al.* (1972).

Micrographs and diffraction patterns were also obtained from detrital mica grains held in a matrix of siderite within a sedimentary carbonate concretion (Sample CHH8, taken from a freshly exposed quarry face of the Hepworth Iron Company at Hazlehead, near Penistone, England). Many similar iron carbonate concretions occur in this sequence of Westphalian A shales. The mineralogy and geochemistry of Concretion CHH8 have been described elsewhere (Curtis *et al.*, 1972; Phakey *et al.*, 1972; and Oertel and Curtis, 1972).

Methods. A powder preparation of a portion of the muscovite crystal, Sample MS 2715, was made by moderate grinding and preparing a smear on a glass plate from a suspension in water. No attempt was made to reduce the pronounced preferred orientation that results from this method.

A fragment of muscovite from the same specimen (not necessarily the same crystal) that had been broken off the specimen was used for a precession camera photograph (W. A. Dollase, personal communication).

For transmission of X-rays a slice of the same crystal that furnished the powder for the powder diffractogram was cut orthogonally to (001) and in a plane approximately parallel to (010). This slice was then reduced by normal thin-section technique to a thickness of approximately 100 μm. At about this thickness the relationship between intensity of reflected beam and absorption is optimal. The slice was then floated off the glass base on which it had been prepared and transferred to the adhesive side of clear adhesive tape, which in turn was mounted taut against an aluminium frame. This frame was held against a 10 mm diameter window in the centre of a 116 mm diameter sample holder, which blocked completely the circular opening in the original Norelco goniometer (fig. 1). Cobalt $K\alpha$ X-radiation was collimated with 1 mm interior diameter baffles and made to impinge on the sample at an angle of $(\pi/2 - \theta)$ with the sample plane. This arrangement shielded the detector almost completely from air and other scatter originating in the X-ray beam before it reached the sample.

After transmission, a much attenuated primary beam continues on a straight-line path from the collimated beam, and a diffracted beam leaves the sample at $(3\pi/2 + \theta)$ with the sample plane whenever a crystal plane is in Bragg orientation. The detector, a scintillometer, is held so that it intercepts this diffracted beam. A tubular radiation shield, constructed like a collimator, is located between the sample and the detector. This almost completely eliminates scatter generated in the primary beam after its transmission through the sample. The radiation shield has a frontal radiation baffle with a 5 mm diameter circular hole, and is terminated toward the detector by a slit 2 mm high (plane of 2θ) and 4 mm wide (plane orthogonal to the plane of 2θ). Behind the window there is an approximately 25 μm thick iron filter which removes the Co-$K\beta$ component.

The apparatus is designed to produce optimal pole figures from polycrystalline rock samples, not for the best possible discrimination of Bragg reflections at different 2θ.

Electron-transparent specimens, oriented to maximize the number of phyllosilicate grains with {001} orthogonal to the specimen plane, were prepared from Samples W22 and CHH8 as described in detail by Phakey *et al.* (1972). Thin sections about 30 μm thick were polished on both sides. After examination under the petrographic microscope, suitable areas for transmission electron microscopy were cored out as discs of about 3 mm diameter. After a sup-

porting 75 mesh copper grid was cemented to them, these discs were floated off the glass slide, thinned into an ultrathin section on the ion beam thinner, and coated with a thin layer of carbon to avoid surface charging during examination in the electron microscope. Occasional thinning of the copper grid to electron transparency provided internal calibration of diffraction patterns.
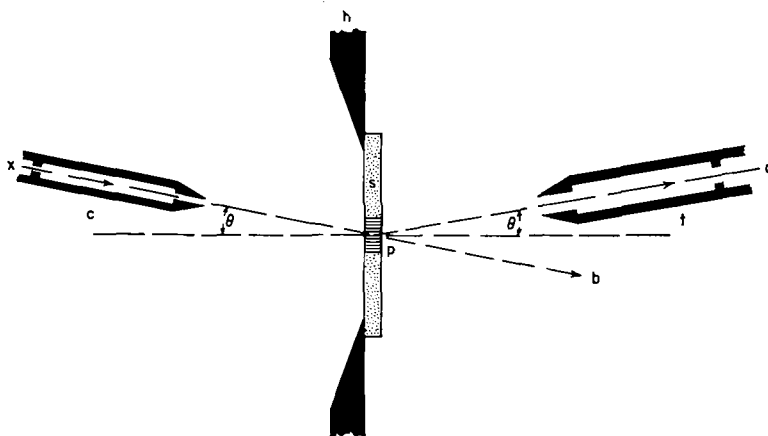

FIG. 1. Modified Norelco pole-figure goniometer. X-rays come from the source —x (not shown), pass through the baffled collimator—c, to the sample—s—where a phyllosilicate grain—p—is in Bragg condition at a given θ. Apart from the direct beam—b, a diffracted beam originates in the grain and passes through a tubular, baffled radiation shield—t—to the detector—d (not shown). The sample covers a window in the holder—h, which can be rotated about the normal to the specimen plane and also tilted about an axis normal to the plane of the drawing. These two rotations are not used when θ is varied systematically in order to produce a diffractogram.

Observations. The powder diffractogram (fig. 2a) of Sample MS 2715 is that expected for a 2M muscovite with the intensity of ool, l even, reflections enhanced as a result of preferred orientation. The reflections 'forbidden' for 2M muscovite (space group C2/c, Henry and Lonsdale, 1952) of ool, l odd, are absent.

A precession camera photograph from a sliver of the same material produced a diffraction pattern with a diffuse streak connecting the ool spots. Along this diffuse streak anomalous maxima were not seen, not even on 36-hour exposure (W. A. Dollase, personal communication).

Transmission X-ray diffractograms of the same single muscovite crystal from Sample MS 2715 that furnished the powder sample are shown on fig. 2b–d. Fig. 2b and c shows the diffractogram of the sample so oriented that the basal plane is in Bragg condition at appropriate angles of 2θ. ool peaks are present for even and for the 'forbidden' odd l. The ool, l even, reflections are broadened in comparison with the powder diffractogram; this is due only to the inferior focusing geometry of the transmission method. The oo1, oo3, and oo5 reflections are also present, successively lower and broader in this order. The 'background' is elevated all along the line connecting the ool reflections, as can be seen by comparing fig. 2b and c with the diffractogram, fig. 2d, made with the basal plane in the plane of θ. This diffractogram
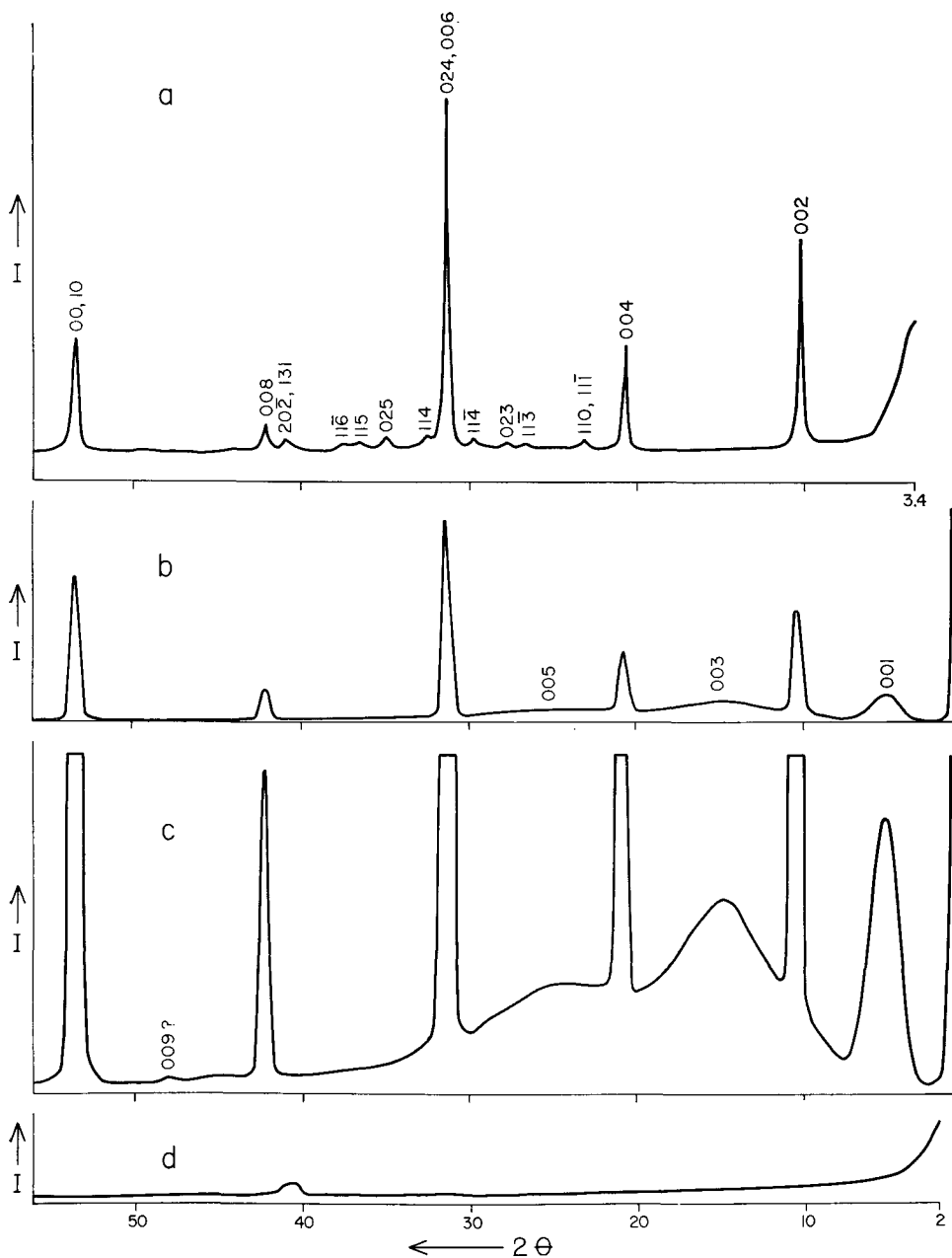
FIG. 2. X-ray diffractograms from portions of one individual muscovite crystal (Sample MS 2715). (a) Powder diffractogram, measured with Cu-$K\alpha_1$ radiation; horizontal (2θ) scale adapted to that of the Co-$K\alpha$ radiation scale of diffractograms (b) to (d); vertical scale, I—intensity in arbitrary units; numbers on the curve—indices of individual reflections. (b) Transmission diffractogram of 100 μm thick slice of the same crystal, normal on (001) bisects angle (π-2θ), and a-axis parallel to goniometer axis. Co-$K\alpha$ radiation. Intensity—I—in arbitrary units. (c) Same as (b), vertically enlarged ten times. (d) Transmission diffractogram of same slice as (b) and (c), so rotated about the specimen-normal that a-axis bisects angle (π-2θ) and normal to (001) parallel to goniometer axis. Vertical scale enlarged ten times with respect to (c).

is ten times enhanced along its vertical scale compared with fig. 2c and a hundred times compared with fig. 2b. The single, small peak at $2\theta = 41°$ is a weak contribution of the $20\bar{2}$ reflection.

In the slate (Sample W22, figs. 3, 6, 8, 9, 10), those numerous phyllosilicate grains that have their basal planes parallel to the incident electron beam, or that can be so rotated, produce selected-area electron diffraction patterns with well-defined 00l rows of spots. From the basal spacing the phyllosilicate mineral phases present can be readily classified as either mica $(d_{002} = 10\cdot0$ Å$)$ or chlorite $(d_{001} = 14\cdot1$ Å$)$. In the siderite concretion (Sample CHH8, figs. 4, 5, 7), fewer phyllosilicate grains are appropriately oriented with respect to the incident electron beam, but those that are can be identified as either mica or kaolinite $(d_{001} = 7\cdot1$ Å$)$ as described previously by Phakey et al. (1972).

Most of the grains in fig. 3a (W22) have their basal planes normal to the specimen plane. Therefore, four rows, A to D, of 00l diffraction spots can be seen distinctly, a fifth, E, faintly, on the selected-area diffraction pattern fig. 3b. The grains by which the diffraction patterns are produced are labelled by the same letters in fig. 3a. From the spacing of spots, flake B can be recognized as chlorite; the others are muscovite. The muscovite patterns show all three principal types found to be characteristic of this mineral in the samples examined. The muscovite grain D has a 'clean' pattern with only the 'allowed' array of spots for 00l, l even, reflections. The apparent broadening of some of the spots is due to partly overlapping spots from other grains. Grain A has a pattern with a streak connecting the 00l spots. Grain C shows, in addition to a similar streak, spots lying halfway between those with even l values. The weak spots are the 00l, l odd, reflections forbidden in $C2/c$ symmetry.
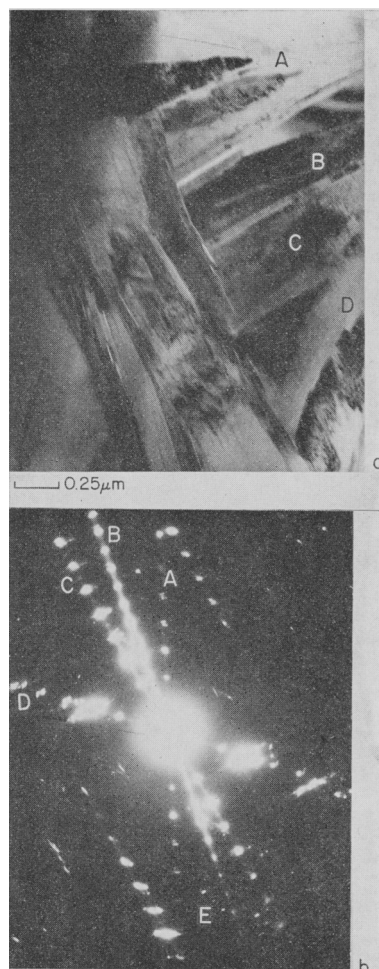

FIG. 3. Transmission electron micrograph (a) and selected-area diffraction pattern (b) of an aggregate of phyllosilicate grains in slate (Sample W22). A, C, D—muscovite; B—chlorite.

Fig. 4 is another example of a fairly 'clean' diffraction pattern from a muscovite flake, without strong streaking or anomalous 00l, l odd, spots. This muscovite grain comes from a sediment (CHH8). The diffraction spots are slightly distorted because flake F is bent. Extensive streaking of 00l spots is found in fig. 5 (CHH8) and fig. 6 (W22). The spots for odd l in the 00l row are weak in fig. 6 but strong in fig. 7 (CHH8).

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Fig. 8a shows a single muscovite grain from the slate (W22) and its diffraction pattern. The basal plane makes an angle of about 30° with the plane of the specimen. The orientation of the grain is shown in the equal-area projection, fig. 8b. Parallel to the trace of the basal plane (TT″ on fig. 8b), fringes due to stacking faults parallel

FIGS. 4 and 5: FIG. 4 (left). Transmission electron micrograph and diffraction pattern of muscovite flake embedded in siderite nodule (Sample CHH8). Pattern of ool spots without streaking and restricted to spots for even l. FIG. 5 (right). Transmission electron micrograph and diffraction pattern of muscovite flake embedded in siderite nodule (Sample CHH8). The diffraction pattern shows a streak connecting the ool spots of flake G. The material surrounding the flake is siderite.

to (001) can be seen on the electron micrograph. These stacking faults are planes across which the orderly stacking sequence of phyllosilicate layers is disrupted and which give rise to characteristic diffraction-contrast fringe patterns. Fringe counts on individual faults are impossible because several faults overlap everywhere in this electron micrograph. This was found to be a general phenomenon in all the phyllosilicate grains in which we could study the fringes, and it is caused by the short distance between stacking faults parallel to (001) on these crystals. The partial dislocations that must of necessity occur where a stacking fault terminates can be observed in fig. 8a; they are labelled D.

Fig. 9 shows an aggregate of phyllosilicate grains in slate (W22) much like that of fig. 3, except that the diffraction pattern reveals that flakes A, B, and C are chlorite ($d_{001} = 14·2$ Å). Row A is 'clean', B and C are streaked. Unlike the case for muscovite, no 'forbidden' spots can be observed in the ool row of chlorite because none are

FIGS. 6 and 7: FIG. 6 (left). Transmission electron micrograph and selected-area diffraction pattern of two muscovite crystals in slate (Sample W22). Grain H shows weak superlattice spots oo*l*, odd *l*, in addition to the stronger normal spots. The larger grain—*K*—has a streaked pattern. FIG. 7 (right). Transmission electron micrograph (*a*) of a portion of large (approximately 7×2 μm) muscovite flake in siderite nodule (Sample CHH8) with selected-area diffraction pattern (*b*). Arrow points to diffraction spots for 'oo7', a superlattice spot.

excluded by the space group $C2/m$ of this mineral (Henry and Lonsdale, 1952). The dark lines seen in grains B, C, and D mark the traces of the stacking faults parallel to (oo1); they result from overlapping diffraction-contrast fringes because the (oo1) planes are nearly parallel to the incident electron beam, and individual fringes cannot be resolved. The average spacing between these stacking faults is approximately 500 to 1000 Å. The somewhat broader lines are due to overlapping and unresolved stacking faults. Some of them may include a large number of stacking faults. Grain A also

contains stacking faults parallel to (001), revealed by the displacement of bend contours where they cross the faults. Compared to fig. 9, especially distinct fringes along widely spaced stacking faults in chlorite can be seen in grain F of fig. 10 because the (001) planes of that grain are favourably inclined with respect to the electron beam.
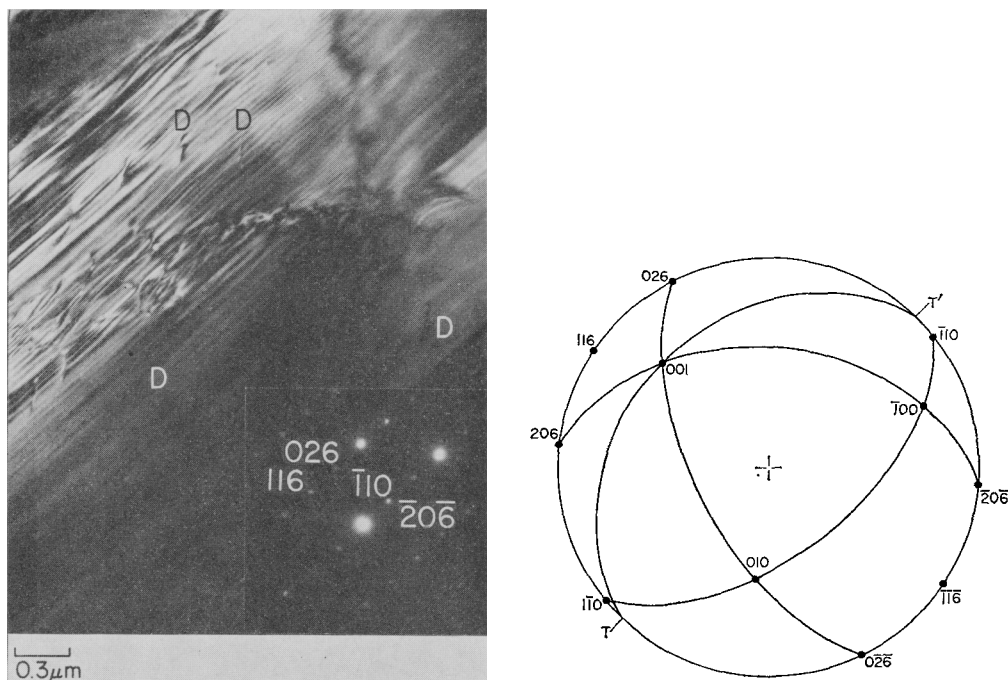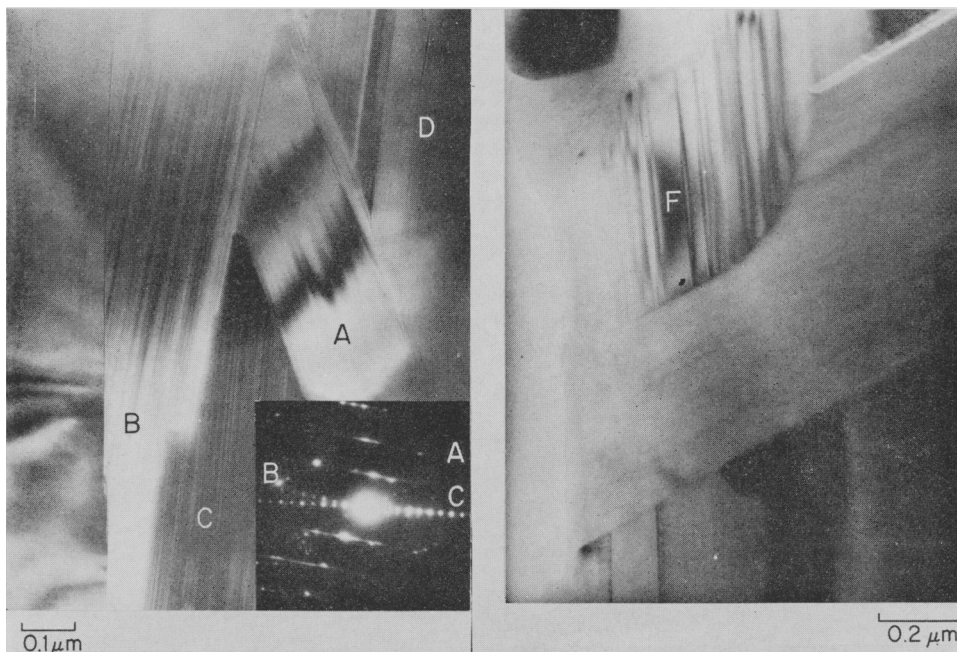

FIG. 8. Transmission electron micrograph (*a*, left) of a muscovite grain in slate (Sample W22) showing fringes on stacking faults parallel to (001) and dislocations—D. Indexed diffraction pattern inserted. Equal-area projection (*b*, right), drawn from the diffraction pattern, shows orientation of the crystal and important crystallographic zones. TT'—trace of fringes in (*a*).

Fig. 11 is a dark-field electron micrograph of a single muscovite grain from the slate (W22) showing fine-scale structures, which appear to be small domains (approximately 100 Å). Similar structures can sometimes be seen in the bright-field (fig. 3, grain C; fig. 6, grain H).

Discussion and conclusions. Various phenomena observed on crystals of muscovite and chlorite are caused by the different ways in which the phyllosilicate layers can be stacked. Stacking is possible with angles of stagger $n\pi/3$ where n is an integer including zero (Smith and Yoder, 1956). In a fully ordered crystal n is constant throughout the crystal or alternates in a short sequence. Widely spaced 'mistakes' in the stacking law (i.e. stacking faults) have little effect on diffraction characteristics of a crystal but could be observed directly under suitable conditions. Such near-perfect crystals produce typical cross-grid electron diffraction patterns with the 00*l* spots forming a row if the (001) plane is parallel, or nearly so, to the incident electron beam. Muscovite with the

space group $C2/c$ should have ool spots only for even l, whereas chlorite with the space group $C2/m$ has the complete set of reflections. Such grains were observed for both kinds of crystals (muscovite, fig. 3, grain D and fig. 4, grain F; chlorite, fig. 9, grain A).

Stacking faults, thin ordered domains, platelets, and thin precipitates that are coherent with the matrix in a crystal can lead to streaking in diffraction patterns because

FIGS. 9 and 10: FIG. 9 (left). Transmission electron micrograph of slate (Sample W22). All grains are chlorite. There are stacking faults parallel to (001) in flakes B, C, and D. The bend contours in grain A are displaced where they cross a stacking fault. Selected area aperture was placed to include grains A, B, and C. ool rows for B and C are slightly streaked. FIG. 10 (right). Transmission electron micrograph of chlorite flakes in slate (Sample W22). Flake F shows fringes on inclined stacking faults.

their presence smears out the reciprocal lattice points in the direction perpendicular to them (Wooster, 1962; Hirsch *et al.*, 1965; Andrews *et al.*, 1967). If the domain has m unit cells along an edge, the lattice points have spikes extending to positions whose coordinates are $\pm\, 1/m$ away from the centre. The intensity of the streaks is, therefore, a measure of stacking disorder. The diffraction patterns of the phyllosilicates reported here show various degrees of streaking (muscovite, fig. 3, grain A; fig. 5; fig. 6, grain K; chlorite, fig. 3, grain B; fig. 9, grains B and C). Since phyllosilicates have layered structures and are known to form numerous polymorphs (Bragg and Claringbull, 1965; Verma and Krishna, 1966), it is reasonable to assume that the streaking of the spots in muscovite is due mainly to the presence of stacking faults and ordered domains.

Electron micrographs of crystals containing stacking faults at an angle with the incident beam are expected to show fringe patterns due to the phase difference α

suffered by electrons on traversing the faults. This phase difference is $\alpha = 2\pi \mathbf{g} \cdot \mathbf{R}$, where $\mathbf{g}$ is the operating diffraction vector and $\mathbf{R}$ is the displacement vector of the fault (Hirsch *et al.*, 1965). Stacking faults on (001) and of average spacing 500 to 1000 Å are directly imaged on chlorite grains (figs. 9 and 10).
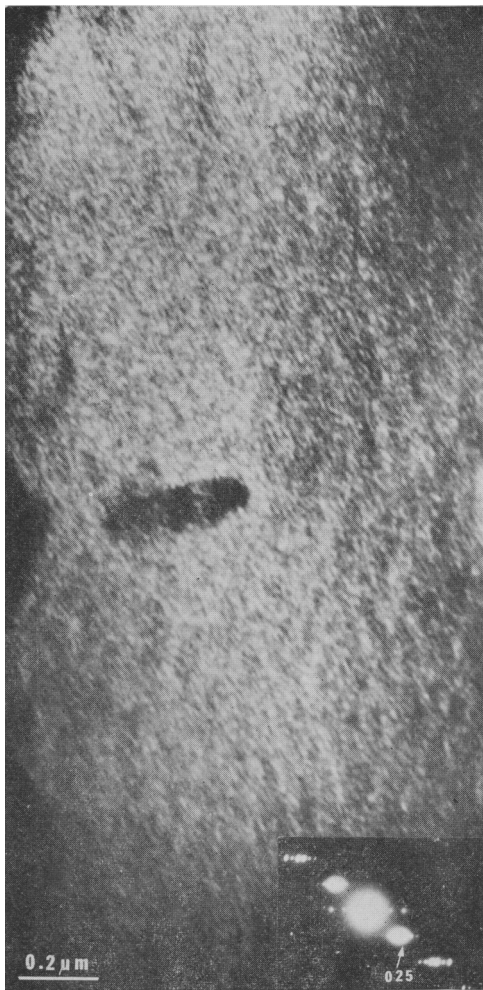

FIG. 11. Ordered domains in a muscovite grain. Dark field micrograph, $\mathbf{g} = 025$, obtained with the Berkeley high-voltage electron microscope.

Distinct fringes are visible on inclined stacking faults in the chlorite grain F of fig. 10, but the (001) planes in other grains of figs. 9 and 10 form too small an angle with the incident beam to show well-resolved fringes. The streaking of the diffraction spots along [001] due to stacking faults on (001) in grains B and C is also observed. The fine-scale lines parallel to the traces of (001) planes, especially clearly seen in grains of figs. 4, 6, and 7, are due to very closely spaced stacking faults in (001) in muscovite. Well-developed fringes are observed in the muscovite grain in fig. 8a in which (001) forms an angle of 60° with the incident beam. The partial dislocations show that some stacking faults are terminated inside the grain. The streaking of the diffraction spots along [001] due to stacking faults on (001) is also observed (fig. 3, grain A; fig. 5; fig. 6).

The nature of the fringes on stacking faults depends on α, with the invisibility criterion for a fringe being $\alpha \pm 2\pi n$, where n is an integer including zero. Accordingly, it is possible, in principle, to find the fault vector $\mathbf{R}$ uniquely from the diffraction contrast. A condition for this, however, is that the projections of fault planes in the plane of the micrograph do not overlap. The fringe patterns of overlapping stacking faults are too complex to allow the calculation of $\mathbf{R}$ uniquely. Unfortunately, stacking faults in both muscovite and chlorite in our samples are always too closely spaced for this purpose. It is apparent, nevertheless, that fringes are invariably absent when faults are so inclined that the operating diffraction vector $\mathbf{g}$ is normal to the trace of the faults. This suggests that the displacement vector $\mathbf{R}$ lies in the plane of the fault.

Superlattice reflections in alloys are now a well-known effect attributed to long-range order. Many examples of long-range order are given in a recent review by Sato and Toth (1965). Superlattice reflections and domains due to Al/Si ordering have been observed inl unar and terrestrial plagioclase (Radcliffe et al., 1970; Christie et al., 1971; Heuer et al., 1972; Hutcheon et al., 1972; Müller et al., 1972; Wenk et al., 1972). More recently, superlattice reflections and domains due to $(NaCl)/(CaCO_3)$ ordering have been observed in scapolite (Phakey and Ghose, 1972). The occurrence of order can have simple geometrical effects on diffraction patterns. For example, given the unit cell of the disordered pattern with systematic absences of reflections, then ordering gives reflections, generally faint, in the forbidden positions (Andrews et al., 1967). By analogy with alloys, plagioclase, and scapolite, it seems reasonable to suggest that the forbidden ool, l odd, reflections in muscovite (fig. 2; fig. 3, grain C; fig. 6, grain H; fig. 7) are superlattice reflections due to ordering. Closely spaced stacking faults having a marked periodicity can result in ordering, thus forming a new and larger unit cell with a symmetry different from $C2/c$. The presence of ordered domains can also contribute to the streaking of the diffraction spots, more or less strong according to domain size (Andrews et al., 1967). This effect can be seen in fig. 3, grain C, and fig. 6, grain H. If ordering involves more than one original unit cell, additional diffraction spots appear at positions corresponding to fractional indices. Since superlattice spots other than ool, l odd, are not observed in muscovite, it seems that unit cells larger than twice the size of the original $C2/c$ cell may not be present. The superlattice reflections in muscovite are usually too weak to reveal the domains in the dark field. However, small (approximately 100 Å) domains have been directly observed in muscovite grains using the Berkeley high-voltage electron microscope (fig. 11). Similar mottled structures can sometimes be seen in the bright field when a systematic row of ool reflections is operating. None of our observations could be taken as evidence for ordering in chlorite.

It remains to explain the absence of superlattice reflections from the powder X-ray diffractogram of the same muscovite crystal as shows them distinctly in a transmission X-ray diffractogram. It is probable that the average path of X-rays through a crystal must be relatively long to detect the long-range order that constitutes the superlattice. X-rays with long paths through powder grains are relatively attenuated by absorption in relation to X-rays diffracted on a path only a few unit cells deep into the body of the crystal. Along such a short path the periodicity of stacking faults that constitutes the long-range order cannot produce an effect that could make a contribution to the diffraction pattern.

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